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Investigation of the effects of Solpawa SBR-X™ doses on total petroleum hydrocarbon in Loam soil at different depths

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ABSTRACT

Laboratory trials of environmental remediation products bring the hope of inventing such products to reality for many of today's intractable environmental pollution conditions, paving the way for the field trials and subsequent large-scale field application. This laboratory study was conducted as the first phase of 2-tiered adaptive trials of Solpawa® Bioremediation System (Solpawa-SBX) funded by the Nigerian Content Development and Monitoring Board (NCDMB). The study investigated the effectiveness of Solpawa-SBX, an indigenously formulated bioremediation agent comprising the liquid Solpawa® soil conditioner (SSC) and granular Solpawa® soil booster (SSB), in the bioremediation of petroleum hydrocarbon-contaminated loam soil. To do this, a total of twelve reactors of $1m^3$ (1000L) each were used (loam soil type and three Solpawa® addition levels). Each reactor was charged with 1000kg of each of the soil types and a condition of heavy spill was simulated by homogenizing the soil with $0.03132m^3$ (31.32L) of crude oil, according to literature. The setup was left quiescent for five (5) days to incubate. The working solutions were prepared by diluting the SSC with water in the ratios of 1:4, 1:6 and 1:8. Each soil type in the reactor was then spiked with each cocktail of the SSC, excluding the soil in the control reactor. This was followed by the addition of $0.004m^3$ (4L) of water after 48 hours and the SSB after another 72 hours. Afterwards, mixing and watering was carried at 2 days interval for a period of 40 days. Selected physiochemical properties of the initial soil conditions and the fertilizer values of the SSC and SSB were determined using standard analytical protocols with regular monitoring and evaluation up until the cessation of treatment. Result showed that the fertilizer values of the SSC and SSB far exceeded the fertilizer value of 3: 2.5: 0.5 (N: P: K) recommended in the literature for materials with remediation potential. The total petroleum hydrocarbon (TPH) of the contaminated soil dropped from an initial 20,406.9mg/kg at commencement of treatment to 681.01mg/kg (representing 96.4% TPH reduction) at the depth of 3 cm after 40 days of treatment with the SSC working solution of 1:4 (i.e., the highest SSC addition level). Overall, TPH reduction across the reactors reduced with increase in depth and decrease in the level of SSC in the working solution. Analysis of variance (ANOVA) results also revealed significance difference in treatment means at 5% significance levels. It is, therefore, suggested that the Solpawa-SBX can be used as

remediation agent for degrading TPH concentration for petroleum contaminated loam soil.

Keywords: Depths, Doses, locally manufactured nutrient, Remediation, Total Petroleum Hydrocarbon.

1. INTRODUCTION

Contamination of the soil environment by hydrocarbons (mostly petroleum hydrocarbons) is becoming prevalent across the globe. This is probably due to heavy dependence on petroleum as a major source of energy throughout the world, rapid industrialization, population growth and complete disregard for environmental health. The amount of natural crude oil seepage into the soil was estimated to be 600,000 metric tons per year with a range of uncertainty of 200,000 metric tons per year (Ayotamuno et al., 2006). Release of hydrocarbons into the environment whether accidentally or due to human activities is a main cause of water and soil pollution. It has been widely reported in the open literature that the causes and effects of crude oil pollution in Nigeria are well documented (Ayotamuno et al., 2006; Akujuru and Ruddock, 2014).

Total petroleum hydrocarbon is referred to small chain alkanes, alkenes and alkynes (C6-C40), for example methane, ethane, propane, etc. Some of the small chain alkanes, such as isopentane, always have a low boiling point of between 60 and 17 (Department of Petroleum Resources, 2002). Since oil and gas became the mainstay of Nigeria's economy (NDES, 1995), the Niger Delta environment has become increasingly vulnerable to oil pollution. Currently, dealing with oil pollution in the Niger Delta has been largely by remediation by enhanced natural attenuation (RENA).

RENA has been reported in several pieces of literature to be ineffective at depths below 1 m due to difficulties with oxygen diffusion beyond 1 m depth of soil. Besides, soil is highly variable in terms of nutrient distribution partly caused by the nutrient imbalance and microbial death created by the contaminating oil (Gomez and Sartaj, 2014). To rejuvenate the microbial population and balance the nutrient distribution in soil, nutrients must be applied within recommended environmental constraints (Hartley et al., 2016). At the moment, there is a dearth of information in the open literature on how to tackle this problem in the Niger Delta without huge financial implications in foreign exchange due to dependence on imported nutrients; therefore, there is a need to evolve a bioremediation nutrient with locally available materials to overcome this challenge (Igoni, 2018). This informed the decision of an indigenous scientist to develop *Solpawa* SBR-X™ in line with the local content initiative of the Nigerian Government. *Solpawa* SBR-X™ is scientifically formulated soil bioremediation system proposed to have the ability to release nutrients into the soil to stimulate microbial activities (Kamath et al., 2004).

However, since its invention, there is no report in the literature of the laboratory or field application of *Solpawa* SBR-X™ to ascertain its effectiveness in decontaminating petroleum hydrocarbon-contaminated soil. This gap led to the commissioning of the Solpawa field and laboratory trials by the Nigerian Content Development and Monitory Board (NCDMB) (Kumar et al., 2011). Therefore, this study aim is to evaluate the effectiveness of Solpawa in the degradation of total petroleum hydrocarbon at different soil depth in loamy soil (Kumar et al., 2011).

2. MATERIALS AND METHODS

Study Area Description

The Niger Delta region of Nigeria is located in the Southern part of the Country and bordered to the South by the Atlantic Ocean and the East by Cameroon. It covers nine states in Nigeria based on political classification. Abia, Akwa-Ibom, Cross River, Edo, Imo and Ondo States were included as part of the Niger Delta region to join the parent Niger Delta namely Rivers, Bayelsa and Delta states. But the core Niger Delta is Delta State (Igoni, 2018). Researchers reported the studies conducted by Niger Delta Development Commission (NDDC), that the region occupies a surface area of about 112,110 square kilometres (National Population Commission, 2006). It represents about 12% of Nigeria's total surface area and has about 450km2 coastlines and is the world's second largest delta and terminates at Imo River. The region as 20,000km2 is the largest of all wetlands in Africa as well as the third largest globally (Okparanma and Mouazen, 2013).

Niger Delta region has three main vegetation namely mangrove forest, fresh water swamp forest and the savanna and stunted rain forests of the sand ridges and mainland margin. Its ecological zone has classified into four namely lowland rain forest, fresh water zone, mangrove swamp and coastal inland zones. The region is classified as tropical rain forest with ecosystem comprising myriads of species of flora and fauna, aquatics and terrestrials (Sojinu et al., 2010). The Niger Delta region accommodates about 23 % of Nigeria population's total population of over 140 (National Population Commission, 2006). The region is the major source of timber and forest products and contains important areas of highly endangered wildlife with very high fishery and agricultural

potentials and also Nigeria as oil and gas producing nation, solemnly depends on the Niger Delta, where most of its oil and gas industries are located.

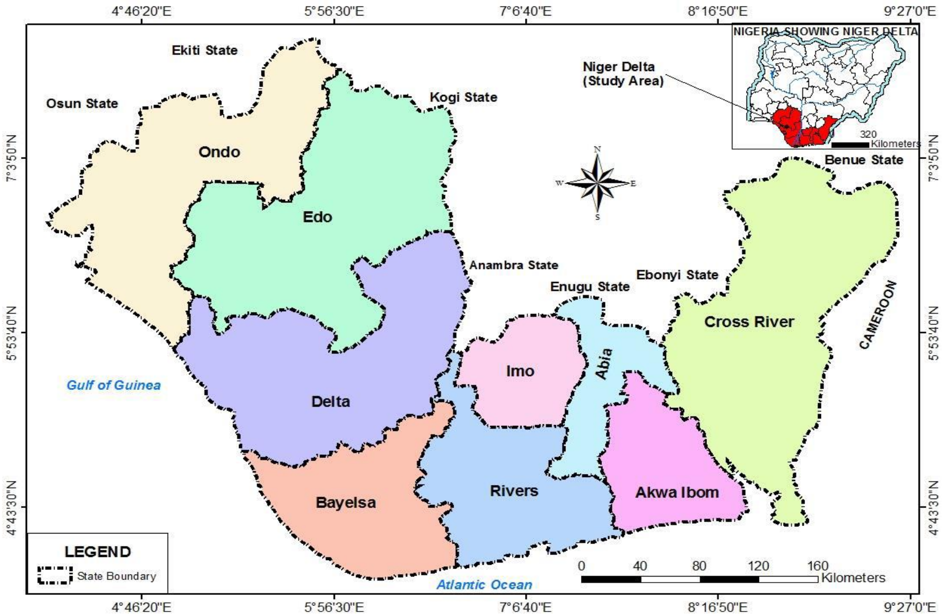

Figure 1 Map of Niger Delta Region of Nigeria Showing States

Sample Collection

For this research, loam soil was collected from Rumuduru in Obiakpo Local Government Areas of Rivers State, in the Niger Delta Region of Nigeria. Crude oil was collected from an oil and gas exploration and exploitation company in the region. The crude oil samples were managed in accordance with the guidelines of the Department of Petroleum Resources (National Population Commission, 2006). The *Solpawa* SBR-X™ was collected from Solpawa Bioremediation Products Company Lagos, Nigeria.

Experimental Design

This experiment was conducted during the peak of raining season in the middle of August to the ending of October 2021. The 3 X 4 factorial experiments in a randomized complete block design (RCBD) were used in this study. The experimental reactor 1 m^3 (1000L) which was be divided into four treatments of 12 reactors each. Randomization was achieved using the draw lot approach as described (Uyigue and Agho, 2007).

Sample Preparation

For the experiment, twelve (12) reactors of $1m^3$ (1000l), loam soil type and three Solpawa® additional levels were used. Each reactor was charged with 1000kg of loam soil and a condition of heavy spill was simulated by homogenizing the soil with $0.03132m^3$ (31.32l) (Ayotamuno et al., 2006). One reactor containing each soil type served as the control. These mixes were in separate reactors for 7 days undisturbed before being taken to the laboratory for analysis. On the 7th day Solpawa Soil Conditioner (SSC) mixed with water at the ratio of 1:4, 1:6 and 1:8 was administered into the contaminated soil in the different reactors with exception of the control. These were allowed for 5 days undisturbed to allow the conditioner to infiltrate into the contaminated soil and samples were collected at different depths of 0.3, 0.6 and 0.9 m from the reactors before taken to the laboratory for analysis. On the 12th day another treatment was introduced to the soil samples in the reactors. This treatment was the addition of Solpawa Soil Booster (SSB) to the reactors at the ratio of 1:12 (20 kg of Solpawa booster and 240 kg of soil). Also, soil samples were collected from the reactors, and they were taken to the laboratory for analysis. Two days after the 12th day, water was applied to the different reactors and samples were collected for laboratory analysis. This was carried out at the interval of 10 days each for 40 days. The physio chemical properties analysed for in the laboratory were total petroleum hydrocarbon (TPH) in crude oil polluted soil respectively on a scale of concentrations in consideration of the range of target and intervention values for TPH in the soil (Department of Petroleum Resources, 2002).

Particle Size Distribution (PSD) and Soil Textural Classification

The hydrometer method was used to determine the soil textural class in the laboratory. It was also determined using soil textural triangle according to United States Department of Agriculture (USDA), soil textural classification. Finally, the textural class was determined using soil triangle according to United State Department Agriculture (USDA) soil textural classification scheme using TAL for Windrows.

TPH Determination

Samples were analysed in line with USEPA 8015 method (USEPA 2003) using Gen Tech master G equipped with a split/split less injector, J and W 30-meter DB-5column and an FID detector. The carrier gas Helium was set at 20ml/min. The injector temperature set at 240℃ and the detector temperature at 300℃. The total run time at 1 NI injection volume through the capillary column with a slow ramp rate 40℃ to 310℃ was 45 minutes. The gas chromatography was calibrated using three working standards prepared from a stock solution of Accu standard Hydrocarbon window defining standard 500mg/ml in chloroform. About 5g of soil sample was used for the analysis. The sample was dried chemically by use of 5g sodium sulphate anhydrous, extraction was conducted separately by using 10ml Dichloromethane and O-Terphenyl and was used as surrogate. Combination of extraction solvent and sample was then agitated by shaking it for 45mins with a vortex mixer and Glass wool/ glass funnel was used to decant extract. The extract was then allowed to concentrate to 1 minute before taken to a gas chromatography for TPH analysis.

Statistical Analysis

A two ways Analysis of variance (ANOVA) was used to determine the significance of the difference in the mean values of each treatment of TPH during the experiment. This was done based on the F-test. Differences were considered significant if $F_{computed} > F_{table}$ at 5% significant levels. The ANOVA was implemented using Microsoft Excel 2019.

3. RESULTS AND DISCUSSION

Soil Textural Classification

Prior to contamination of each of the soils, soil samples were collected using soil corer for particle size distribution (PSD) analysis, from the three different soils studied in this research. The result of the uncontaminated soils indicated the relative content of soil particles of various sizes such as sand, silt and clay in the soil (Table 1). From the results, it was revealed that the soil textures were Loam according to United State Department Agriculture (USDA) textural classification of soil using TAL for windows a software used by the USDA to classify soil types (Figure 2).

Table 1 Particle Size Analysis Results

Sand %	Silt %	Clay %	Textural Class
38.60	36.20	25.20	Loam
90.60	5.40	4.00	Sand
5.60	31.09	63.31	Clay

Fertilizing Value of Solpawa Conditioner and Booster

The initial NPK characteristics determined were used as indices for evaluation of the fertilizing value (i.e., remediation potential) of the Solpawa products. Table 2 shows the NPK values of the Solpawa conditioner and booster. As in Table 2, the Solpawa booster (granular form) had a higher fertilizing value than the conditioner (liquid form). Compared with the recommended fertilizer value of 3: 2.5: 0.5 (N: P: K) for materials, both the booster and conditioner far exceeded these typical values. This suggests that both Solpawa booster and conditioner are excellent candidates for remediation purposes, which justifies the use of the products for remediation works.

Table 2 NPK Values of Solpawa Conditioner and Solpawa Booster

Properties	Solpawa Conditioner	Solpawa Booster
N (mg/kg)	103.560	261.450
P (mg/kg)	1.241	7.592
K (mg/kg)	180.903	569.191

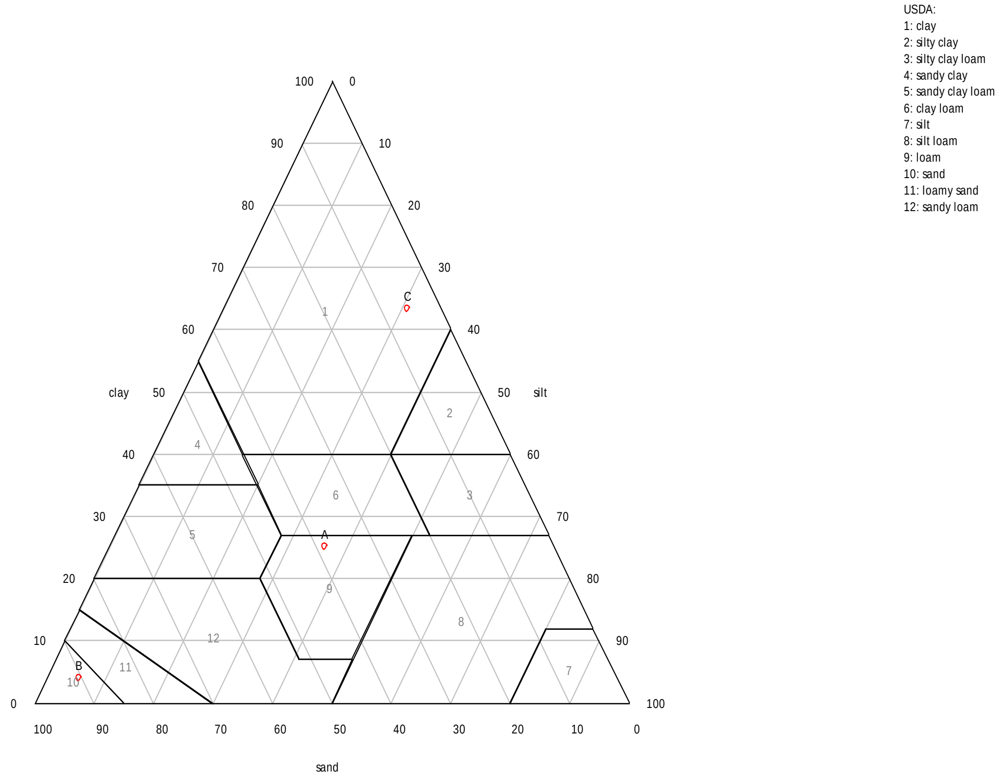

Figure 2 USDA Soil Texture Triangle

Effect of Different Doses Solpawa on TPH at 30 cm Soil Depth

The effect of Solpawa doses on TPH concentration at 30 cm depth in Loam soil (Table 3). Also, Figure 3 shows the differences in TPH concentration in Loam soil with time at 30 cm soil depths at different doses of Solpawa. An examination of the Figure 3 showed that the TPH concentrations decreased with reduction in Solpawa conditioner to water ratio administered to the contaminated Loam soil over time and also in presence of oxygen. It might be so because at the depth of 30 cm, the presence of oxygen speeds up the degradation of TPH more than it does at depths where oxygen is less in supply. The TPH reduced by 3.00, 10.00, 9.00 and 8.00% for the depth of 0.3 m in T_1, T_2, T_3 and T_4, respectively (Table 4). The TPH concentrations contained in the reactors ranged from 18335.60 to 2065.72 mg/kg at the depth of 30 cm for 7 days after contamination, 12 days after Solpawa conditioner (ASC), 10, 20, 30 and 40 days after Solpawa booster (DSB), in T_1, T_2, T_3 and T_4 respectively according to their degradation rate (Table 3). TPH percentage reduction due to the presence of Solpawa conditioner and booster was significant (Table 4). These ranged from 0 to 96.4 % in T_1, T_2, T_3 and T_4 at the depths of 30 cm for day 7, 12 ASC, 10, 20, 30 and 40 DSB, respectively (Table 4). This might be due to the Solpawa conditioner and booster. The ANOVA result for the effect of different doses of Solpawa conditioner and boaster on the TPH concentration revealed that there was significant difference in the treatment means at 5% significance levels. This suggests that with 95% confidence, the difference in treatment means was due to the difference in Solpawa conditioner and booster applied.

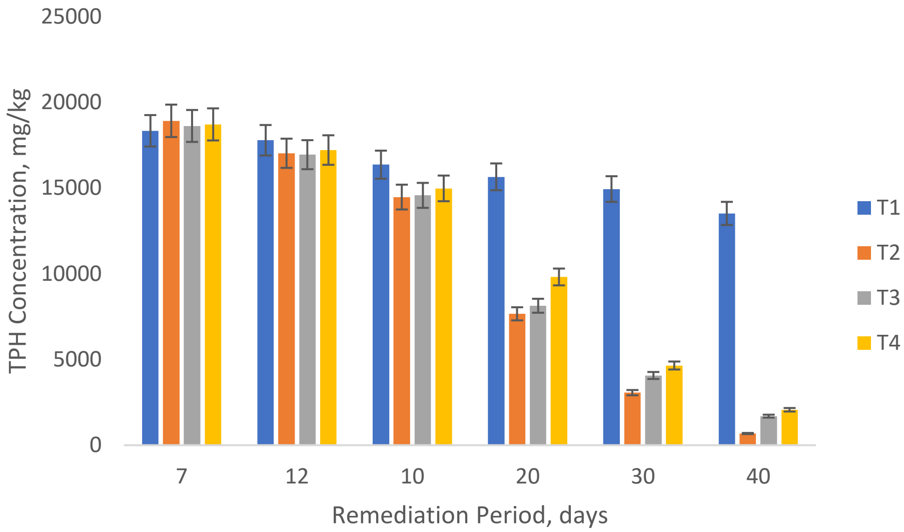

Figure 3 Effect of Different Doses of Solpawa on 30 cm Soil Depths

Effect of Different Doses Solpawa on TPH at 60 cm Soil Depth

Figure 3 shows the disparity of TPH concentration in loam soil with time at depths of 60 cm. Also, the effects of Solpawa doses on the variation in TPH concentration and days of remediation in loam soil is (Table 3). An inspection of Figure 4 showed that the TPH concentrations decreased with reduction in Solpawa conditioner -to- water ratio applied to the contaminated loam soil over time. It also decreased slightly with increase in depth of 60 cm compared to 30 cm depth. It might be so because at the depth of 60 cm, the presence of oxygen lessens the speeds up the degradation of TPH more than it does at depths where oxygen is more in supply. The TPH reduced by 2.00, 9.00, 8.00 and 7.00% for the depth of 60 cm in T_1, T_2, T_3 and T_4, respectively (Table 4). The TPH concentrations contained in the reactors ranged from 19251. 76 to 3471.55mg/kg at depth of 60 cm for 7 days after contamination, 12 days after Solpawa conditioner (ASC), 10, 20, 30 and 40 days after Solpawa booster (DSB), in T_1, T_2, T_3, and T_4 respectively according to their degradation rate (Table 3). TPH percentage reduction due to the presence of Solpawa conditioner and booster was significant (Table 4). These ranged from 0 to 90.8999 % in T_1, T_2, T_3 and T_4 at the depths of 60 cm for day 7, 12 ASC, 10, 20, 30 and 40 DSB, respectively (Table 4). This might be due to the Solpawa conditioner and booster. The ANOVA result for the effect of different doses of Solpawa conditioner and boaster on the TPH concentration has shown that there was significant difference in the treatment means at 5% significant level. This suggests that with 95% confidence, the difference in treatment means was due to the difference in Solpawa conditioner and booster applied.

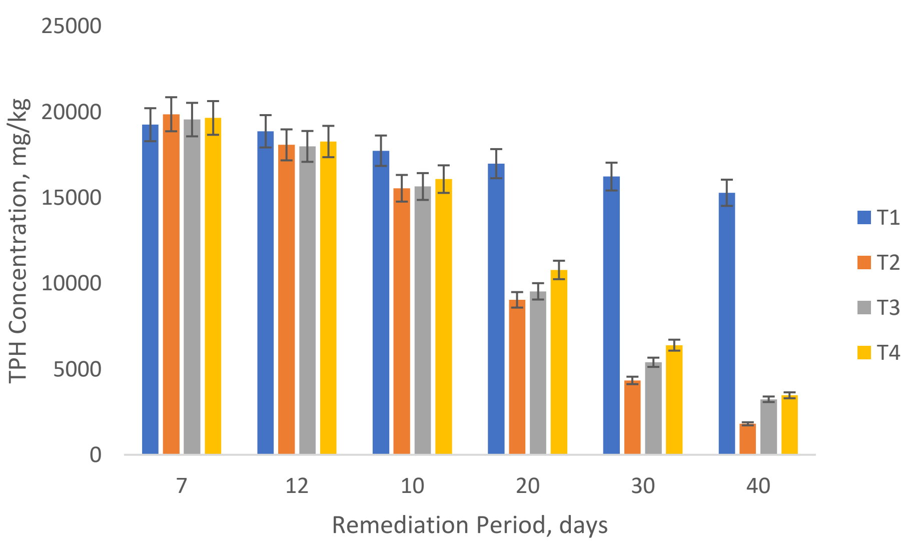

Figure 4 Effect of Different Doses of Solpawa on 60 cm Soil Depths

Effect of Different Doses Solpawa on TPH at 90 cm Soil Depth

The changes caused by the effects of Solpawa doses on TPH concentration with reference by days of remediation in loam soil (Table 3). Also, Figure 5 displays the change of TPH concentration in loam soil with time at different depth of 90 cm). Figure 5 also displayed that the TPH concentrations decreased with reduction in Solpawa conditioner to water ratio administered to the contaminated loam soil over time. It also slightly reduced with increase in depth of 90 cm compare to 30 and 60 cm depths, respectively. It might be so because at the depth of has lesser oxygen present that slow the speeds up the degradation of TPH more than it does at depths where oxygen is more in supply. The TPH reduced by 0.90, 8.00, 7.00 and 5.00% for the depth of 90 cm in T_1, T_2, T_3 and T_4, respectively (Table 4). The TPH concentrations contained in the reactors ranged from 21054.61 to 3680.35mg/kg at the depth of 90 cm for 7 days after contamination, 12 days after Solpawa conditioner (ASC), 10, 20, 30 and 40 days after Solpawa booster (DSB), in T_1, T_2, T_3 and T_4 respectively according to their degradation rate (Table 3). TPH percentage reduction due to the presence of Solpawa conditioner and booster was significant (Table 4). These ranged from 0 to 82.51998% in T_1, T_2, T_3 and T_4 at the depths of 90 cm for day 7, 12 ASC, 10, 20, 30 and 40 DSB, respectively (Table 4). This might be due to the Solpawa conditioner and booster. The ANOVA result for the effect of different doses of Solpawa conditioner and boaster on the TPH concentration has shown that there was significant difference in the treatment means at 5% significant level. This suggests that with 95% confidence, the difference in treatment means was due to the difference in Solpawa conditioner and booster applied.

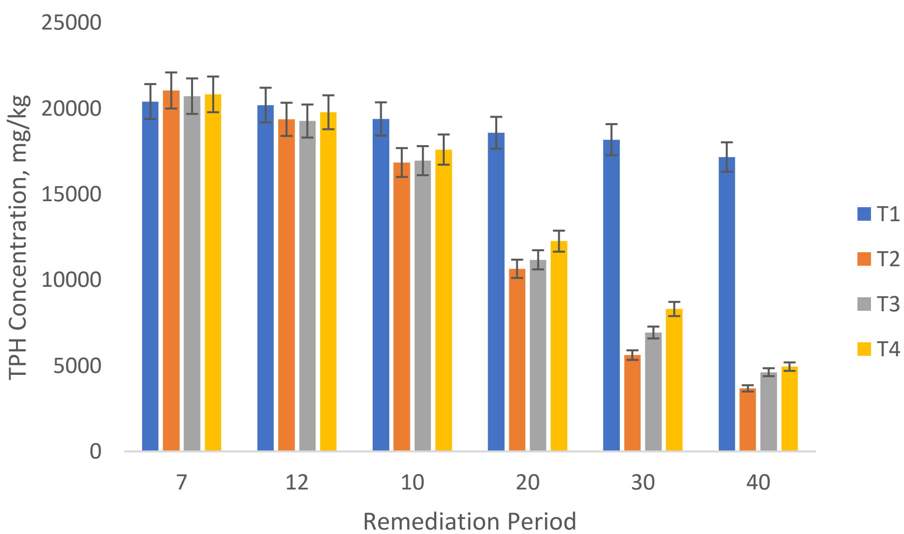

Figure 5 Effect of Different Doses of Solpawa on 90 cm Soil Depths

Table 3 Mean Results of TPH (mg/kg) Concentration in Contaminated Loam Soil Treated with Solpawa

Period	T_1			T_2			T_3			T_4		
(days)	d_1	d_2	d_3	d_1	d_2	d_3	d_1	d_2	d_3	d_1	d_2	d_3
7	18335.6	19251.8	20406.9	18918	19862.8	21054.6	18620	19549.9	20723	18710	19646.5	20825.4
12	17785	18866.7	20202.8	17025.3	18075.2	19370.3	16943.3	17986	19272.3	17214.1	18271.3	19784.1
10	16362.2	17734.7	19394.7	14471.5	15544.7	16852.1	14571.2	15647.8	16959.7	14976.3	16078.7	17607.8
20	15650.8	16980.1	18586.6	7661.39	9037.6	10653.6	8132.78	9532.55	11178	9812.05	10780.1	12266.1
30	14939.4	16225.4	18182.5	3064.55	4338.05	5617.37	4066.39	5395.79	6938.04	4647.81	6394.95	8309.31
40	13516.6	15282	17172.4	681.01	1807.52	3680.35	1694.33	3237.47	4625.36	2065.69	3471.55	4946.02

$T_{1,2,3,4}$ = Treatment, $d_{1,2,3}$ = depths (30, 60 and 90 cm)

Table 4 TPH Concentrations Percentage Reduction (%) in Contaminated Loam Soil Treated with Solpawa

Period	T_1			T_2			T_3			T_4		
(days)	d_1	d_2	d_3	d_1	d_2	d_3	d_1	d_2	d_3	d_1	d_2	d_3
7	0	0	0	0	0	0	0	0	0	0	0	0
12	3.003174	2.000025	0.999958	10.00476	8.999972	7.999958	9.004887	7.999973	7.000062	7.995083	6.99996	5.00006

10	10.76294	7.880022	4.959952	23.50402	21.73999	19.95995	21.7442	19.95996	18.16005	19.95575	18.15994	15.45006
20	14.64277	11.80001	8.919946	59.50211	54.49996	49.39997	56.32234	51.24	46.06002	47.5572	45.12998	41.10005
30	18.52266	15.72002	10.89997	83.80088	78.15997	73.32	78.16117	72.39997	66.52003	75.15869	67.44999	60.10002
40	26.28242	20.62004	15.84997	96.4002	90.89999	82.51998	90.90048	83.44	77.68002	88.95943	82.32997	76.25

$T_{1,2,3,4}$ = Treatment, $d_{1,2,3}$ = depths (30, 60 and 90 cm)

4. CONCLUSIONS

The fitness of Solpawa conditioner and Solpawa booster for bioremediation of total petroleum hydrocarbon contaminated loam soils was investigated using experimental methods. Results support the following conclusions: It was revealed that the soil textures were loam, sand and clay according to the United State Department of Agriculture (USDA) textural classification of soil. Solpawa conditioner and booster have the potential for remediation of petroleum hydrocarbon contaminated soils because of their fertilizer (NPK) values far exceeded the recommended value of 3: 2.5: 0.5 (N: P: K). Solpawa conditioner and booster influenced the biodegradation of TPH, in the hydrocarbon petroleum contaminated loam soil. The Solpawa conditioner degraded TPH up to 10%, after 12 days of remediation for the depths of 30, 60 and 90 cm. In addition, Solpawa booster further degraded TPH up to 96.40%, after 40 days of remediation. ANOVA results showed that there was significant difference among the treatments at different depths (30, 60 and 90 cm).

Informed consent
Not applicable.

Ethical approval
Not applicable.

Conflicts of interests
The authors declare that there are no conflicts of interests.

Funding
The study has not received any external funding.

Data and materials availability
All data associated with this study are present in the paper.

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